Available online at www.scholarsresearchlibrary.com

Scholars Research Library

Archives of Applied Science Research, 2014, 6 (4):157-161
(http://scholarsresearchlibrary.com/archive.html)

ISSN 0975-508X
CODEN (USA) AASRC9

Growth and characterization of L-histidine doped copper sulphate crystals

S. Mary Delphine[1], R. Krishna Priya[2], T. Lidia Arockia Thai[1] and S. Ajitha[1]

[1]Department of Physics, Holy Cross College, Nagercoil
[2]Department of Physics, Rajas International Institute of Technology for Women, Nagercoil

---

ABSTRACT

Single crystals of pure and L-histidine doped Copper sulphate have been grown by slow evaporation of an aqueous solution at room temperature. The grown crystals were subjected to powder X-ray diffraction analysis, confirming that the crystalline nature of the crystal. UV-Vis-NIR spectra have been recorded in the range 190-1100 nm. The microhardness test was carried out in plane and the hardness coefficient was calculated.

Key words: Crystal growth, X-Ray diffraction, L-Histidine, Copper Sulphate, UV, Microhardness.

INTRODUCTION

Crystal growth from solution is a very important process that is used in many applications from the laboratory to the industrial scale. An innumerable number of organic and inorganic crystals are grown in this fashion [1]. Organic crystals have large nonlinear susceptibilities compared to inorganic crystals. Most organic NLO crystals have usually poor mechanical and thermal properties and are susceptible to damage during processing [2]. Several compounds related structurally to the amino acid histidine have been studied by diffraction techniques [3-7]. L-histidine salts have received much attention not only due to their unique properties but also for possible applications to noval photonic and optoelectronic devices [8]. The crystals were grown by slow evaporation technique [9-10]. The grown crystals are subjected to powder X-ray analysis, UV-Vis NIR and the Microhardness measurements and the results are discussed [11].

MATERIALS AND METHODS

2.1 Crystal Growth

Analytical Reagent (AR) grade of pure and L-histidine doped Copper sulphate in different molar ratios such as 0.01, 0.03, 0.05, 0.07 and 0.09 was grown. The mixture was placed in a magnetic stirrer and stirred for about 3 hours. Long time stirring was done for the homogenous mixing of the mixture. In the solution growth technique, selection of a solvent which is moderately soluble plays an important role [12]. In the present study deionised water was used as the solvent for synthesis of the pure and L-histidine doped Copper sulphate crystals. The solution was optimally covered using a plane thin plastic sheet. Crystals of pure and L-histidine doped Copper sulphate were grown from its saturated aqueous solution by the slow evaporation technique within 20 days. Transparent good quality crystals with perfect shape and free from macro defects were used for characterization.

3. Characterization Studies

The grown crystals of pure and L-histidine doped Copper sulphate crystals were analysed by powder XRD analysis. Powder XRD analysis was carried out to study the crystallinity by using an X-ray diffractmeter, MODEL RICH, SEIFERT, XRD 3000 P , with monochromatic nickel filtered CuKα (λ= 0.15406 nm) radiation. The sample was scanned over the range 10 - 70^0 at the rate of 1^0/min. The linear optical properties of the crystals were examined between 200 and 2000 nm using the VARIAN CARY 5E UV- Vis – NIR spectrophotometer. Microhardness studies have been carried out on pure and L-histidine doped Copper sulphate crystal using a MVCCD-1000 Video Digital microhardness tester fitted with a Vickers diamond pyramidal indentor attached to an incident light microscope.

RESULTS AND DISCUSSION

4.1. Powder X-RAY diffraction analysis

The freshly ground powder sample of pure and L-histidine doped Copper sulphate was subjected to powder X-ray diffraction analysis. X-ray diffraction technique is used to investigate the inner arrangement of atoms or molecules in a crystalline material. The lattice parameters are identified by X-ray powder diffraction technique. The recorded PXRD pattern of pure and L-histidine doped Copper Sulphate is shown in figs 1.1, 1.2, 1.3, 1.4, 1.5 and 1.6. The observed sharp peaks confirm the crystalline nature of the grown pure and L-histidine doped Copper sulphate crystals. From the PXRD diffraction data, it is observed the pure and L-histidine doped Copper Sulphate crystals are Anorthic. The crystal data of the pure and L-histidine doped Copper Sulphate crystals are presented in the Table 1, the lattice parameters, the PXRD data have good agreement with the JCPDS value. When, the Lattice parameters changes depand on the dopant concentration.

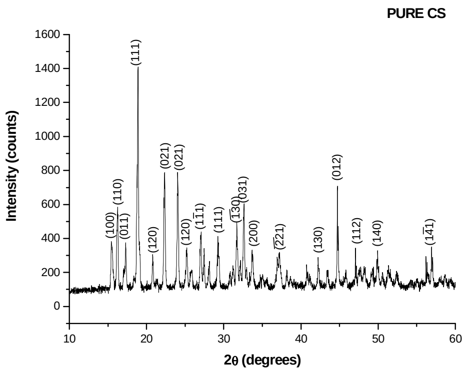

Fig 1. 1. PXRD pattern of Pure CS

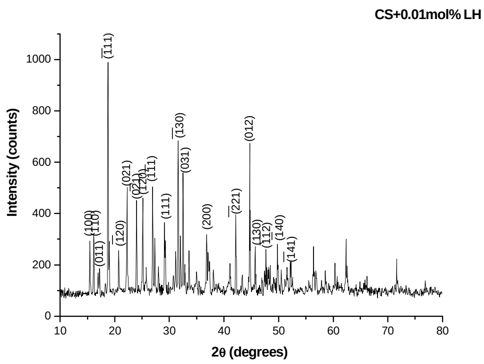

Fig 1. 2. PXRD pattern of 0.01mol%

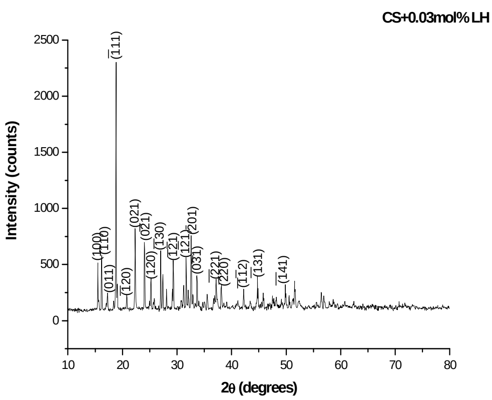

Fig 1. 3. PXRD pattern of 0.03 mol%

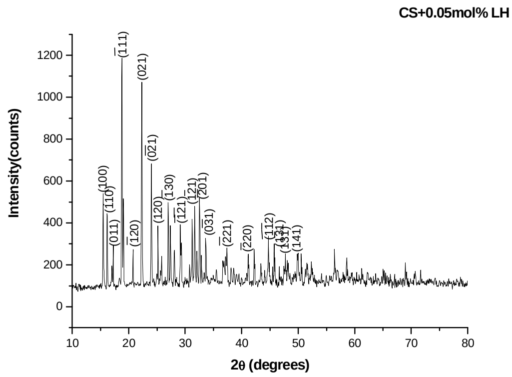

Fig 1. 4. PXRD pattern of 0.05 mol%

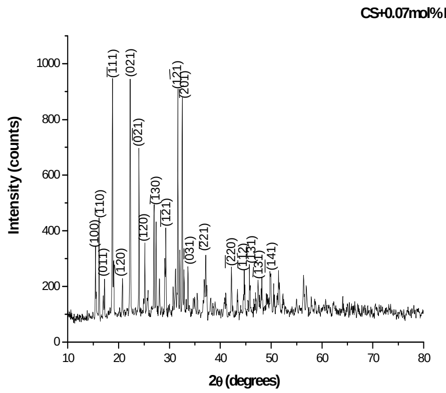

Fig 1. 5. PXRD pattern of 0.07 mol%

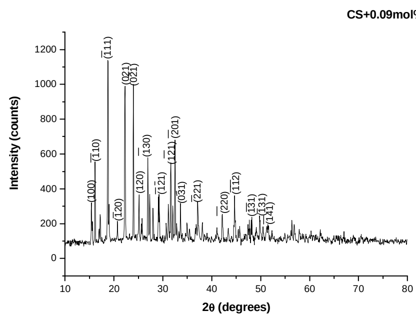

Fig 1. 6. PXRD pattern of 0.09 mol%

Table.1 Lattice parameters of the grown Crystals

system	Pure CS	Concentration of LH in CS					
		0.01 mol%	0.03 mol%	0.05 mol%	0.07 mol%	0.09 mol%
a(A^0)	6.122	6.126	6.208	6.110	5.999	5.992
b (A^0)	10.720	10.542	10.609	10.672	10.335	10.54
c (A^0)	5.968	5.980	6.063	5.963	5.932	5.944
Volume $(A^0)^3$	364.01	492.46	364.71	361.54	341.89	351.20
α	82.350	81.291	81.681	82.455	80.323	82.034
β	107.33	108.23	110.34	107.40	107.84	107.06
γ	102.60	101.64	102.36	102.36	100.42	100.84

4.2 Optical transmission studies

Fig 2 show the UV-Vis NIR spectrum of L-histidine doped Copper sulphate recorded 190 nm to 1100 nm. The absorbance and transmittance spectrum obtained from the Figs. 2, 3. The UV cutoff wavelength is found to be less than 250 nm. The cut-off wavelength for the pure and L-histidine doped Copper Sulphate crystals are almost same. The transmittance is minimum in the wavelength region between 600 to 1100 nm. The transmittance is maximum (100%) in the wavelength region between 320 to 600 nm.

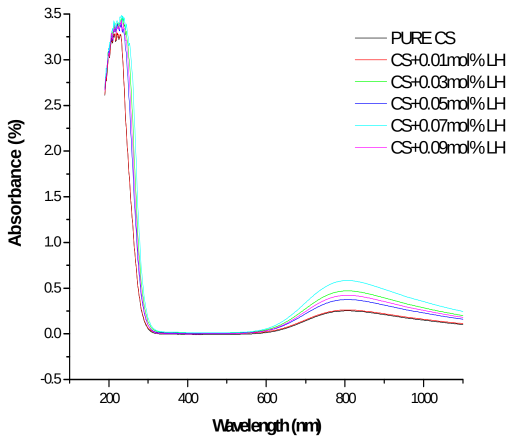

Fig 2: Absorbance spectra of Pure and LH doped CS crystals

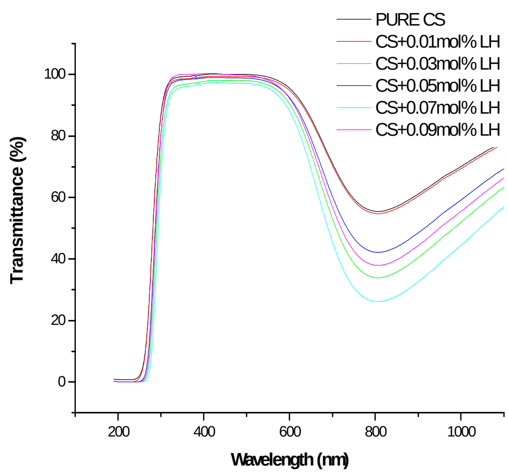

Fig 3: Transmittance spectra of Pure and LH doped CS crystals

4.3 Measurement of Microhardness

The hardness of a material depends on different parameters such as lattice energy, debye temperature, heat of formation and interatomic spacing [13-15]. According to Jianghong Gong , during an indentation process the external work applied by the indentor is converted into a strain energy component proportional to the volume of the resultant impression and a surface energy component proportional to the area of the resultant impression [16]. Microhardness is a general microprobe for assessing the bond strength, apart being a measure of bulk strength [17]. Measurement of hardness is an useful nondestructive testing method to determine the bond strength [18]. The crystal slices are well polished to avoid surface defects which influence the hardness values strongly. Pure and L-histidine doped Copper sulphate crystals were placed on the platform of the microhardness tester and loads of different magnitudes (25, 50 & 100) were applied over a fixed interval of time (10s). The maximum applied load was restricted to 100gm as micro cracks were developed at higher loads. The hardness was calculated using the relation [19].

$$H_v = 1.8544 \frac{P}{d^2} \text{ Kgmm}^{-2}$$

Where P is the applied load in Kg and d is the average diagonal length of the indentation impression in micrometer. The relation between hardness number (H_v) and load (P) for L-histidine doped Copper sulphate crystals are shown in the Fig 3. The indentation hardness is measured as the ratio of applied load to the surface area of the indentation [20-21]. It is observed from the graph that the hardness value increases with increasing load. The increase of the microhardness with the increasing load is in agreement with the normal indentation size effect (ISE). According to Onitsch, 1.0≤n≤1.6 for hard materials and n>1.6 for soft materials [22]. So L-histidine doped Copper sulphate crystals also soft materials.

Table 4: Hardness number (H_v) and load (P) of the grown crystals

P (gm)	Hv Kg/**mm^2**					
	Pure CS	0.01mol% LH	0.03mol% LH	0.05mol% LH	0.07mol% LH	0.09mol% LH
25	33.9	40.02	41.285	43.14	44.36	62.91
50	47.55	50.48	48.81	51.69	59.37	78.31
100	71.15	65.65	68.36	74.97	84.79	83.98

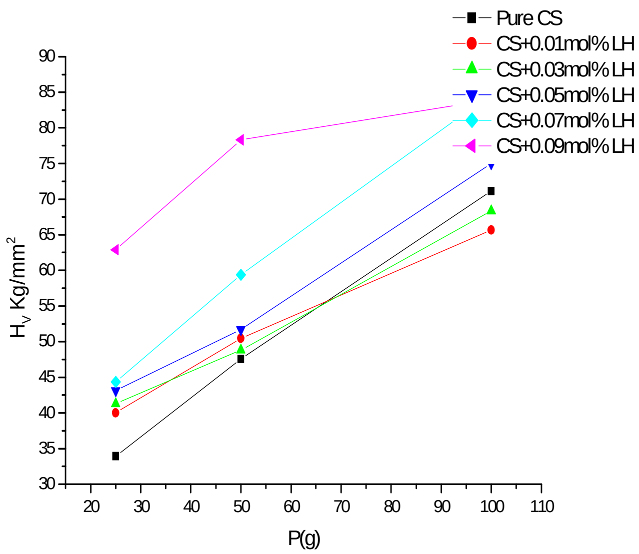

Fig 3. load P (g) versus H_v (Kg /mm^2)

Table 5: Microhardness- Workhardening coefficient

No	Sample	Work hardening coefficient (n)
1.	Copper sulphate	2.34
2.	L-H (0.01 mol%) doped CS	3.21
3.	L-H (0.03 mol%) doped CS	2.36
4.	L-H (0.05 mol%) doped CS	1.63
5.	L-H (0.07 mol%) doped CS	3.27
6.	L-H (0.09 mol%) doped CS	3.54

CONCLUSION

Optically clear, single crystals of pure and L-histidine doped Copper Sulphate are conveniently grown by slow solvent evaporation technique. The single crystals PXRD studies confirm the Anorthic structure of the crystals. UV – Vis NIR spectrum of L-histidine doped Copper Sulphate crystals recorded it is not a transparent crystal but defect free single crystals. The Microhardness study reveals the crystals are soft in nature.

REFERENCES

[1] S.A. de Vries, P. Goedtkindt, W.J. Huisman, M.J. Zwanenburg, R. Feidenhans'l, S.L. Bennett, D.M. Smilgies, A. Stierle, J.J. De Yoreo, W.J.P. Van Enckevort, P. Bennema, E. Vlieg, *J. Crystal Growth* 205 (**1999**) 202.
[2] S.Ajitha, R. Krishna Priya, T. Lidia Arockia Thai, S. Mary Delphine, J. *Archives of Physics Reaserch*, Accepted **2014** APR 187.
[3] H.O. Marcy, M.J. Rosker, L.F.Warren, P.H. Cunningham, C.A. Thomas, L.A. Deloach, S.P. Velsko, Ebbers, J.H. Liao, M.G. Konatzidis, *Opt. Lett*., 20 (**1995**) 252.
[4] H.W. Zhang, A.K. Batra, R.B.Lal, *J. Crystal Growth* 137 (**1994**) 141.
[5] C.C. Frazier, M.P. Cockerham, *J. Opt. Soc. Am.*, B4 (**1987**) 1899.
[6] Duorong Yuan, Zhenwu Zhong, Mingguo Liu, Dong Xu, Qi Fang, Yonghong Bing, Suoying Sun, Minhua J. *Crystal Growth* 186 (**1998**) 240.
[7] Y.J. Ding and X. D. Mu and X.Gu,J.*Nonlinear optical Physics and Material* 9, (**2000**) 21.
[8] S.Aruna, G.Bhagavannarayana, P. Sagayaraj *J. Crystal Growth* 304, (**2007**) 184-190.
[9] X. W. Wang, D. Xu, Lu, D.R. Yuan, and S .X .XU. Silversitein, F.X. Webster, Spectroscopic Identification of Organic Compounds, sixth ed., Wiley, New York, **1998**.
[10] S.Ajitha, R. Krishna Priya, T. Lidia Arokia Thai, S. Mary Delphine, J. *Archives of Physics Research*, 5 (2) (**2014**) 9-13.
[11] T. Dammak N. Fourati, Y. Abid, H. Boughzala, A. Mlayah, C. Minot, *Spectrochimica Acta* A66 (**2007**) 1097.
[12] D. Arivouli, R. Fornari and J. Kumar, *J. Mater. Sci. Lett*., 10 (**1991**) 559.
[13] J. Benet Charles and F. D. Gnanam, *J. Mater. Sci. Lett.*, 9 (1990) 165.
[14] Elsamma Chacko, J. Mary Linet, S. Mary Navis Priya, C. Vesta, B. Milton Boaz and S. Jerome das, *Indian J Pure and Appl.Phy.*,44 (**2006**) 260.
[15] Jianghong Gong, *J. Mater. Sci. Lett*., 19 (**2000**) 515.
[16] S. Mary Navis Priya, J. Mary Linet, R. Robert, C. Vesta and S. Jerome Das J. Loyola *Inst. Frontier Energy* 8 (**2006**) 29-32.
[17] B. W. Mott, Micro indentation hardness testing (**1956**).
[18] P. H. Soni, C.F. Desai and S.R. Bhavasar, *Indian J. Pure and Appl*., 42 (**2004**) 371.
[19] P. Sumithraj, Prem Kumar. X. Sahaya Shajan and H. Alex Devadoss *Indian J. Science and Technology* vol 3 (**2010**) 253-256.
[20] K. G. Subhadra, K. Kishan Rao, and D.B. Sirdeshmukh, *Bull. Mater.Sci*.23, (**2000**) 147.
[21] S. Mukerji and T. Kar, *cryst .Res .Technol*. 34, (**1999**) 1323.
[22] M. Hanneman,*Metall.manchu* 23 (**1941**) 135.